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Characterization and modeling of PEEK in histories with reverse loading

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Abstract

Traditional viscoelastic models for describing polymer response during large deformations are normally designed to capture the response during monotonic loading and typically have difficulty capturing the response after a reversal of the deformation process. In particular, most models pay little attention to capturing the equilibrium stress, the anisotropy developed after plastic flow in the elastic response, and the characteristics of the yield and subsequent flow after reversal of the loading. To characterize these events, the thermo-mechanical response of PEEK is studied during shear histories that have one or more points at which the strain rate is reversed. In particular, using digital image correlation (DIC) methods, the response of PEEK is captured during processes that subject the material to histories that reverse the straining direction one or more times. These studies show that the response of PEEK in monotonic loading is very different from that observed after reversing the loading, and also from that observed in further cycling. Yet, after multiple cycles of loading and reverse loading, if the loading is then continue beyond the point that loading reversal was initiated in the cycling, the response after this point returns to that of the initial monotonic loading.

Keywords: Poly-ether-ether-ketone (PEEK), shear, reverse loading, cyclic loading, plastic flow

1. Introduction

Polyether ether ketone (PEEK) is becoming a favorite material for many high-end applications due to its exceptional thermo-mechanical performance. The mechanical behavior of this material has been extensively characterized by conducting conventional monotonic testing, such as monotonic compression and tension [1]. Most often, constitutive models for PEEK and other polymers are established based on the results of these monotonic loading tests [2-6]. However well these models capture the monotonic loading, a lack of information on how the material behaves once the loading is reversed leaves these models untested for their accuracy in predicting the response under more complex loading histories, particularly ones that include segments of reverse loading. To better understand the response to such loading, to verify the accuracy of existing models, and to motivate potential changes to future models, it is desirable to characterize the response of PEEK and other polymers to tests that have non-monotonic segments.

Cyclic uniaxial loading that takes samples, for example, from tension to compression during large deformation is in general difficult to perform due to a number of issues including gripping and deformation localization, such as necking or shear banding. In spite of this difficulty, there has been an effort to simulate load reversal processes by sequentially testing the response in two different tests such as by first pulling a sample in tension and then testing the response in compression [7, 8]. However, for polymers and other materials that exhibit time dependent response, this sequential testing introduces discontinuous loading history that might miss important relaxations that should be captured in modeling. There are efforts to capture the effects of loading reversal in steels by plane shearing [9], in-plane cyclic compression-tension at small strains [10], and cyclic torsion [11]. The gripping during shearing, for example developed for a single shear sample [12], seems to be an ideal method to study complex loading histories that include large reversals and cyclic loading.

To capture the characteristics of the response of PEEK during reverse and cyclic loading, a double shear sample was developed and tested under different loading histories. It became immediately clear that the initial response during the loading was significantly different from the response in the subsequent reverse or cyclic loading. Yet, once the loading was continued beyond the maximum limit of initial loading, the yielding occurred at essentially the same level, with flow tracking the continuation of the initial monotonic loading response. On examination of the sample, it was noted that the shearing occurred without the development of any localization, such as shear banding, both during monotonic loading [13] and during reverse loading and thus suggests using this sample for studies of the response to complex loading profiles.

2. Materials and Methods

The experiments were conducted on samples cut from an as-received 3/4 inch thick 12x12 inch² sheet of VICTREX 450G PEEK. No additional thermal conditioning was conducted on the samples. Bars of dimension 45x19.05x7 mm³ were cut from this sheet and two slots of $L_o = 2.54$ mm width were milled into each bar to form a double shear sample, as shown in Fig. 1. The thickness of the shearing zone in the sample was 1.91 mm and the two notches were separated by 13.72mm. The samples were mounted in a double shear grip system that fixed the two ends of the sample to the load-frame crosshead and the center of the sample to the actuation piston of an MTS 8500 machine. The shearing strain γ was measured by digital image correlation (DIC) system (Aramis, 2M) using black and white speckles on the flat size of the sample. The speckle sizes were selected to cover 5~12 pixels of the DIC image.

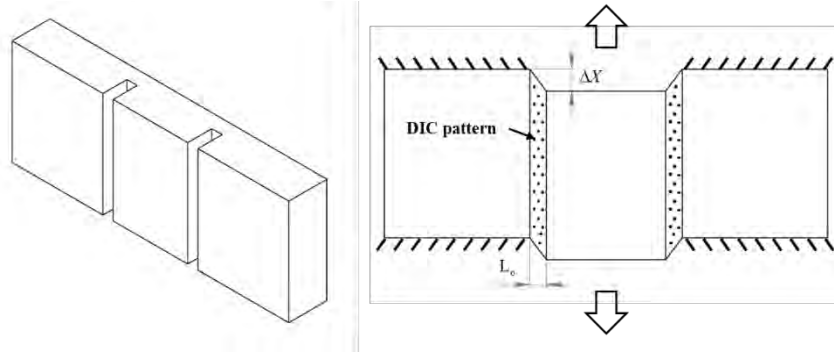


Fig. 1. Schematic of double shear sample (left) and loading process (right)

Two kinds of complex loading are demonstrated using this sample. Fig. 2a shows the first loading path that included an initial segment of cyclic loading with a maximum shear strain $\gamma_{\max} = 0.05$ followed by a second segment of cyclic loading with maximum strain of $\gamma_{\max} = 0.2$. In each segment of the loading or its reversal, the strain rate as measured by the DIC was constant and approximately 0.001 s^{-1} . Fig. 2b shows the second loading path that sequentially increases the limit strains in cyclic loading by 10% shear strain in each cycle up to $\gamma_{\max} = 0.4$. The strain rate, measured by DIC, in each segment is approximately 0.002 s^{-1} .

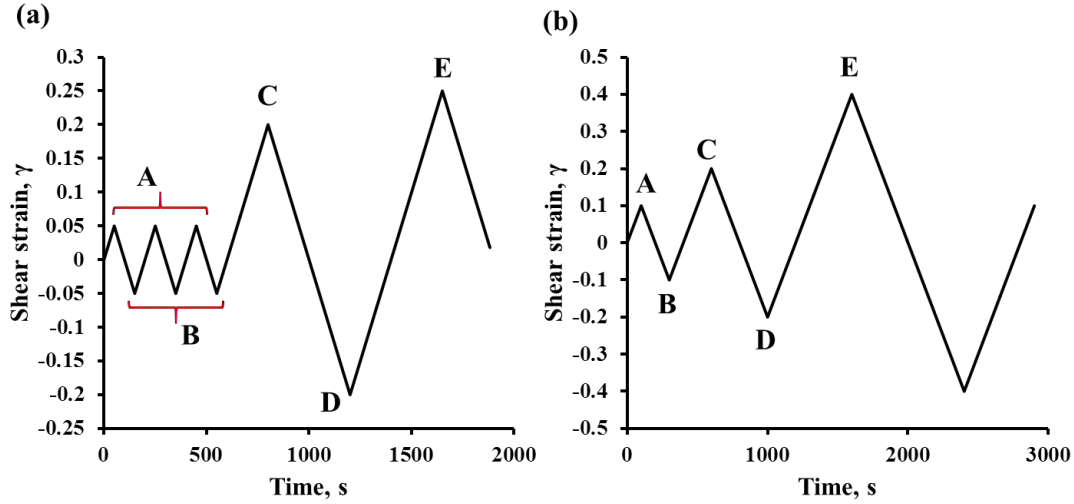


Fig. 2. Strain path for the loading: (a) multiple cycles of small-strain shearing followed by cycles of large-strain shearing, (b) consecutively increasing shearing cycle amplitudes

3. Experimental Results and Discussion

As is described in [12], during deformation one might observe the development of shear bands, particularly in shear loading. The samples were scratched with axial lines and the shearing monitored for the development of shear bands along the sample, as indicated in Fig. 3 for a monotonic loading rate of 0.01 s^{-1} . No such bands developed during the reported tests. During monotonic loading, initial yield occurred at approximately 8% shear strain and then steady flow with a shallow hardening occurred starting at approximately 67MPa. There was no observed softening of the material after the initial yield. At around 40% strain, strain hardening increased slightly. Comparing with the the response of PEEK in compression [1], the initial yield stress is around 50% of the that in compression for the same loading rate. As was observed in tension, compression, and now in shear, in monotonic loading initial yield follows by a very shallow steady flow hardening.

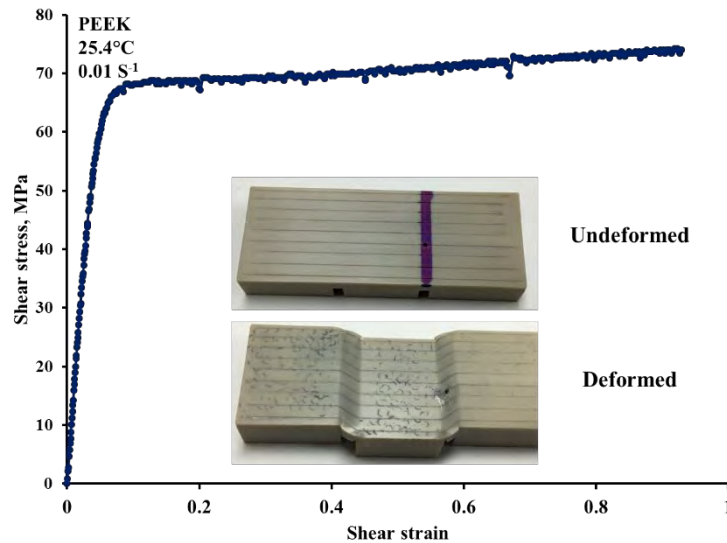


Fig. 3. Monotonic shearing of PEEK

The response of PEEK to the shearing histories described in Fig. 2a and 2b are given, respectively, in Fig. 4 and Fig. 5. As can be seen in Fig. 4, the yield stress in both loading and reverse loading are the same and around 67MPa. In the initial small cycle loading between the limits shown in Fig. 2a as points A and B, the flow is minimal with a

narrow hysteresis loop that crosses zero at approximately 1% and -1.5% strain. Once the larger strain amplitude cycles starts, one sees initial yield and then flow similar to monotonic loading, followed by a transition to a large hysteresis loop (between point C, D and E). In particular, on the initial yielding one sees flow at practically constant stress, while this constant stress flow is not seen during the following cycle (D to E).

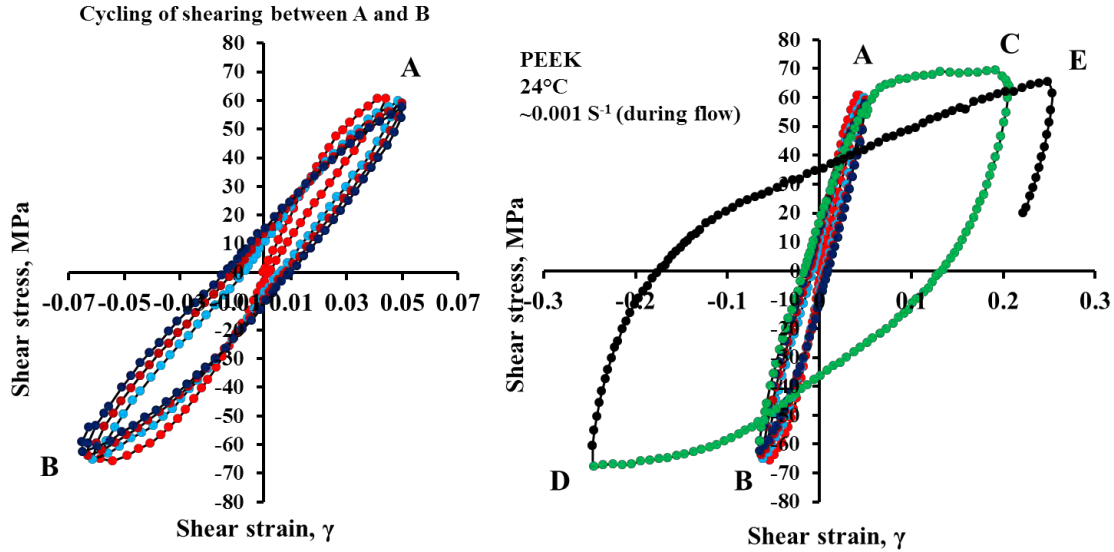


Fig. 4. Mechanical response of PEEK with respect to cyclic shear as shown in Fig. 2a

As shown in Fig. 5, as we sequentially increase the cycle maximum strain, in each cycle one first sees yield and steady flow at about a constant stress of 67 MPa after yielding (approaching points A, C, and E on the figure). Yet, on the reversal of load in the segment, approach to the opposite point (points B, D, and G) occurs without showing a pronounced range of steady constant stress flow, but rather a yielding at, for example F, followed by a steady finite hardening flow, for example from F to D*, until which the flow approaches steady flow at constant stress of 67 MPa. In particular, as the amplitude of the cycle becomes larger, the axis of the hysteresis loop rotates, yet all the responses pass through a node at about 38 MPa and one node at about -42 MPa, consistent with what would be expected from a changing (rotating) kinematic hardening axis [14]. Unlike monotonic shear, which would show constant shear flow passing through points A, C, and E in the figure, cyclic shear shows yield, for example at F, followed by steady flow with substantial hardening, for example F to G. This suggests the presence of two yield mechanisms, one associated with initial yielding and one associated with reverse loading.

The response suggests that the maximum previous strain, once we pass the initial yield, controls the orientation of the back stress axis, while cyclic deformation within this limit does not substantially reorient this axis. In addition, reaching the maximum previous strain is accompanied by steady flow at approximately constant stress (a very shallow hardening) that is accompanied by reorientation of the back-stress axis. This reorientation may be associated with softening of the back stress, possibly through a detangling mechanism, similar to that proposed for some explanations of the Mullin's effect [15].

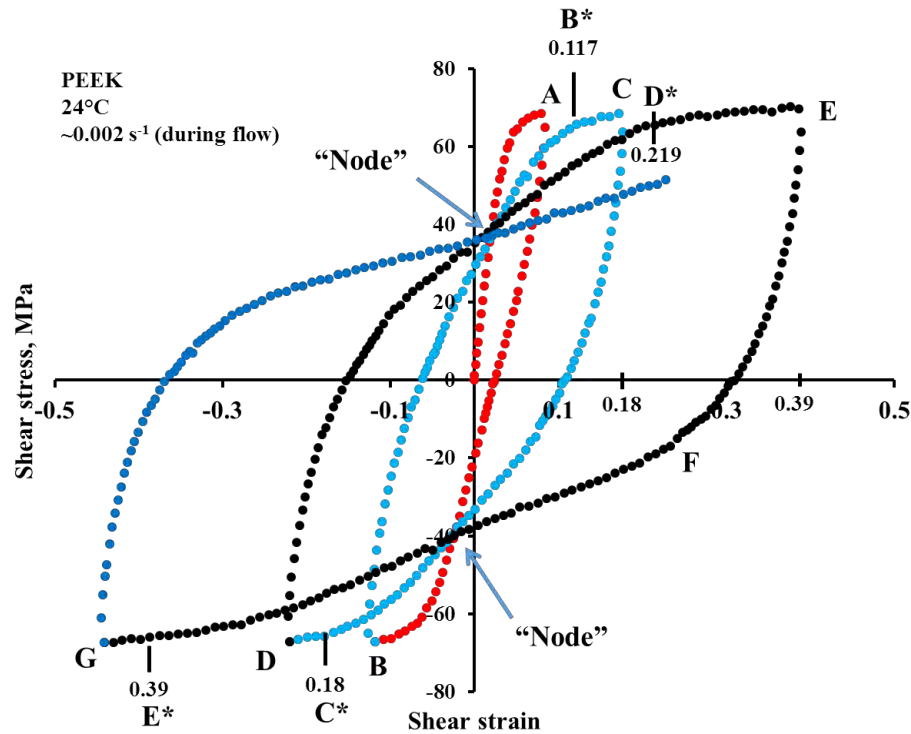


Fig. 5. Mechanical response of PEEK to increasing the cycle amplitude, as shown in Fig. 2b

4. Summary

The response of PEEK in histories with reverse loading has been characterized by conducting reversed cyclic shearing. The response shows close to steady constant stress flow with a very shallow hardening at the limits of both loading and reverse loading, indicating a steady flow mechanism responsible for increasing the previous maximum strain. Yet, cyclic loading within the bounds of the previous maximum strain show hysteresis loop response characteristic of kinematic hardening, with yield followed by steady finite hardening flow parallel to the back-stress axis. The increase of the previous maximum strain occurs with flow at close to constant stress accompanied by a reduction in the kinematic hardening slope, which may be interpreted as a softening of the kinematic hardening.

The response under monotonic and reverse loading, in combination, indicate that there are two flow mechanisms controlling the response of PEEK.

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6. References

1. Rae, P.J., E.N. Brown, and E.B. Orler, *The mechanical properties of poly(ether-ether-ketone) (PEEK) with emphasis on the large compressive strain response*. Polymer, 2007. **48**(2): p. 598-615.
2. Boyce, M.C., D.M. Parks, and A.S. Argon, *Large inelastic deformation of glassy polymers. part I: rate dependent constitutive model*. Mechanics of Materials, 1988. **7**(1): p. 15-33.
3. Arruda, E.M., M.C. Boyce, and R. Jayachandran, *Effects of strain rate, temperature and thermomechanical coupling on the finite strain deformation of glassy polymers*. Mechanics of Materials, 1995. **19**(2-3): p. 193-212.

4. Krempf, E. and C.M. Bordonaro, *A state variable model for high strength polymers*. Polymer Engineering & Science, 1995. **35**(4): p. 310-316.
5. Shim, J. and D. Mohr, *Rate dependent finite strain constitutive model of polyurea*. International Journal of Plasticity, 2011. **27**(6): p. 868-886.
6. Garcia-Gonzalez, D., R. Zaera, and A. Arias, *A hyperelastic-thermoviscoplastic constitutive model for semi-crystalline polymers: Application to PEEK under dynamic loading conditions*. International Journal of Plasticity, 2017. **88**: p. 27-52.
7. Senden, D.J.A., J.a.W. van Dommelen, and L.E. Govaert, *Strain hardening and its relation to Bauschinger effects in oriented polymers*. Journal of Polymer Science Part B: Polymer Physics, 2010. **48**(13): p. 1483-1494.
8. Brown, E.N., et al., *Soft recovery of polytetrafluoroethylene shocked through the crystalline phase II-III transition*. Journal of Applied Physics, 2007. **101**(2): p. 024916.
9. Choi, J.S., et al., *Measurement and modeling of simple shear deformation under load reversal: Application to advanced high strength steels*. International Journal of Mechanical Sciences, 2015. **98**: p. 144-156.
10. Yoshida, F., T. Uemori, and K. Fujiwara, *Elastic-plastic behavior of steel sheets under in-plane cyclic tension-compression at large strain*. International Journal of Plasticity, 2002. **18**(5-6): p. 633-659.
11. Morrow, J., *Cyclic Plastic Strain Energy and Fatigue of Metals*, in *Internal Friction, Damping, and Cyclic Plasticity*, B. Lazan, Editor. 1965, ASTM International: 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. p. 45-45-43.
12. G'Sell, C. and A.J. Gopez, *Plastic banding in glassy polycarbonate under plane simple shear*. Journal of Materials Science, 1985. **20**(10): p. 3462-3478.
13. Li, W., et al., *Mechanical Characterization and Preliminary Modeling of PEEK*, in *Mechanics of Composite and Multi-functional Materials, Volume 7*, C. Ralph, et al., Editors. 2016, Springer International Publishing. p. 209-218.
14. Negahban, M., *The Mechanical and Thermodynamical Theory of Plasticity*. 2012: CRC Press.
15. Maiti, A., et al., *Mullins effect in a filled elastomer under uniaxial tension*. Physical Review E, 2014. **89**(1): p. 012602.